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NATIONAL DIOXIN STUDY TIER 4—COMBUSTION SOURCES

Quality Assurance Evaluation

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1.0 INTRODUCTION

1.1 Introduction

This final report summarizes quality assurance (QA) support provided by Research Triangle Institute (RTI) to Tier 4 of the U.S. Environmental Protection Agency's (EPA's) National Dioxin Study. The work was performed under the following EPA Contract Numbers:

- 68-03-3149, WA 10-1
  (August 16, 1984 through June 30, 1985)
- 68-02-3002, WA 31
  (July 1, 1985 through October 31, 1985)

The EPA Work Assignment Officer was Mr. William B. Kuykendal, and the RTI Project Leader was Richard V. Crume. Mr. Crume was assisted during field and laboratory audits by RTI's Donna J. Holder, Doris J. Smith, and Robert S. Wright.

The purpose of this report is to present an overview of RTI's activities and to summarize RTI's conclusions and recommendations. The report is divided into sections on field audits, laboratory audits, performance audits, and the review of documents. Specific details regarding the activities discussed in each section can be found in the references listed at the end of this report. EPA has requested that the identities and addresses of the industrial facilities involved in the field audits remain confidential. Thus, these facilities are referred to only as Sites ISW-A, BLB-B, and MET-A.

While this report describes RTI's involvement in Tier 4 of the National Dioxin Study, it should be noted that two other organizations also provided some degree of QA support to the study. Radian Corporation performed a number of internal QA activities, including several audits of their sampling and analytical programs.[1] Similarly, EPA's "Troika" Laboratories (i.e., the three dioxin analytical facilities at: Research Triangle Park, North Carolina: Bay St. Louis, Mississippi; and Duluth, Minnesota) established QA requirements for laboratory analyses and data review.[2,3] Although the EPA Work Assignment Officer, William B. Kuykendal, coordinated QA activities for the Tier 4 Study, he remained completely independent of the conclusions and recommendations presented in this and earlier RTI reports.

1.2 Background

In 1984 EPA implemented a national strategy to study the nature and extent of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2378-TCDD) contamination in the environment. The strategy, known as the National Dioxin Study, established seven categories of investigation, ranging from the most probable contamination to the least probable. These categories are as follows:

- <u>Tier 1</u>: 2,4,5-trichlorophenol (245-TCP) production sites and associated waste disposal sites.
- <u>Tier 2</u>: Sites (and associated waste disposal sites) where 245-TCP was used as a precursor to make pesticidal products.
- <u>Tier 3</u>: Sites (and associated waste disposal sites) where 245-TCP and its derivatives were formulated into pesticidal products.
- <u>Tier 4</u>: Combustion sources.
- <u>Tier 5</u>: Sites where pesticides derived from 245-TCP have been and are being used on a commercial basis.
- <u>Tier 6</u>: Certain organic chemical and pesticide manufacturing facilities where improper quality control on certain production processes could have resulted in the formation of 2378-TCDD contaminated product waste streams.
- <u>Tier 7</u>: Control sites where contamination from 2378-TCDD is not suspected.

The responsibility for Tier 4 of the National Dioxin Study was assigned to EPA's Office of Air Quality Planning and Standards (OAQPS). OAQPS's contractor for the Tier 4 sampling and analytical activities was Radian Corporation of Research Triangle Park. North Carolina. Additionally. EPA's Troika Laboratories performed some of the analyses. The Tier 4 activities performed by these two organizations, including the sampling and analytical techniques used, are summarized below.

RADIAN CORPORATION

- Stack Gas Sampling

  Dioxins and Precursors: Using Modified Method 5 Sampling Trains.

  HCl: Using HCl Train (Modified Version of Method 5).

  CO, CO_2, O_2 NO_x, SO_2, THC: Using Continuous Emission Monitors.

- Ambient Air Sampling

  Dioxins and Precursors: Using Ambient XAD Train.

- Process Samples and Data

  Feed Materials and Supplementary Fuels: Using Grab Samples.

  Ashes, Liquors, and Other By-Products: Using Grab Samples.

  Process Data: Taken from Control Room.

- Soils

  Dioxins and Precursors: Using Grab Samples.

- Analyses

  Dioxin Precursors: By GC/MS.

  HCl: By Ion Chromatography.

  Chlorine in Fuel Oil: By Parr Bomb/Ion Chromatography.

- Analyses

Dioxins and Furans: By HRGC/HRMS.

RTI was contracted by OAQPS to provide QA support to the above sampling and analytical activities. Although RTI's support focused primarily on the procedures followed by Radian Corporation, the Troika Laboratories were evaluated to a limited extent through the submission of several performance evaluation samples. RTI's QA support activities can be categorized as follows:

- "Technical Systems Audits" of Field and Laboratory Operations.
- "Performance Evaluation Audits" of Field and Laboratory Operations.
- "Reviews of Quality Assurance Project Plans" and Other Documentation.

A "technical systems audit" is defined as a qualitative, on-site evaluation of a measurement system.[4] The objective of a technical systems audit is to assess and document the use of all: (1) test facilities and associated apparatus; (2) measurement systems; (3) recordkeeping and data validation procedures; (4) equipment operating, maintenance, and calibration procedures; (5) reporting requirements; and (6) quality control procedures. Since the above items were defined in Radian's approved Quality Assurance Project Plan and Site-Specific Test Plans, these Plans provided the basis for RTI's audits. Additionally, RTI prepared and used during the audits a series of check lists reflecting the required standard operating procedures for the methods in use. The results of RTI's technical systems audits are presented in Chapters 2.0 and 3.0.

A "performance evaluation audit" is defined as a quantitative evaluation of a measurement system.[4] Ordinarily, the quantitative evaluation of a measurement system involves the measurement or analysis of a reference material having associated with it a known value or composition. It is important that the value or composition of the reference material be certified, or at least verified, prior to use, and that the certification or verficiation be adequately documented. Usually the identity of the reference material is disguised so that the operator or analyst will treat it no differently from a test program sample. Although it is possible for each measurement of a test program to be subjected to a performance audit, it is more common for only the critical, or most important, measurements to be evaluated. RTI's performance evaluation for the Tier 4 study examined the following systems:

- o <u>Dioxin Analyses</u>: Using four 2378-TCDD audit samples obtained from the National Bureau of Standards.
- o <u>Chlorine in Fuel Oil Analyses</u>: Using a set of four, and a second set of two, No. 2 fuel oil samples spiked with methylene chloride. These audit samples were prepared and verified by RTI.
- o <u>HCl Analyses</u>: Using actual HCl train impinger water verified by RTI.
- o <u>Dry Gas Meter Calibrations (i.e., Sampling Train Meter Boxes) Using a calibrated orifice supplied by EPA.</u>
- o <u>Computerized Calculations</u>: Using a set of verification data supplied by RTI.

The results of RTI's performance evaluation audits are presented in Chapter 4.0.

The "review of quality assurance project plans" involves the evaluation of the plans relative to a set of established criteria. The criteria used by RTI in evaluating several drafts of Radian's Quality Assurance Project Plan are presented in Table 1.[5] In addition to the review of these plans, RTI evaluated the QA and quality control (QC) procedures presented in several Site-Specific Test Plans. The criteria listed in Table 1 were also used in evaluating these Test Plans, although the criteria were less rigorously applied. The results of RTI's evaluation of the Quality Assurance Project Plans and Site-Specific Test Plans are presented in Chapter 5.0.

TABLE 1. CRITERIA FOR EVALUATING QUALITY ASSURANCE PROJECT PLANS

Project Description
Project description
Experimental design
Intended use of acquired data
Start and completion dates
Appropriate diagrams, tables, and figures
Project Organization and Responsibility
Organization of project
Line of authority
Key individuals (including quality assurance official)
Quality Assurance Objectives for Measurement Data
Precision
Accuracy
Completeness
Representativeness
Comparability
Sampling Procedures
Sampling site selection
Sampling procedures
Description of containers for sample collection, preservation, transport, and storage
Procedures to avoid sample contamination
Sample preservation methods and holding times
Procedures for recording sample history, sampling conditions, and analyses to be performed
Sample Custody Records
Preparation of reagents or supplies associated with sample
Location and conditions where sample was taken
Sample preservation methods
Labeling
Field tracking forms
Field and laboratory sample custodians
Laboratory custody log
Laboratory handling, storage, and dispersement procedures

TABLE 1. (CONTINUED)

Calibration Procedures

- Description of, or reference to, calibration procedure
- Frequency of calibration
- Calibration standards, including sources and traceability procedures

Analytical Procedures

- Analytical procedure
- Appropriateness of method

Data Reduction, Validation, and Reporting

- Data reduction scheme
- Equations to be used
- Validation procedures
- Identification and treatment of outliers

Internal Quality Control Checks

- Replicates
- Spiked samples
- Split samples
- Control charts
- Blanks
- Internal standards
- Zero and span gases
- Quality control samples
- Surrogate samples
- Reagent checks
- Calibration standards and devices

Performance and Systems Audits

- Schedule for conducting audits
- Systems to be audited
- Sources of audit materials

Procedures to Assess Data Precision, Accuracy, and Completeness

- Central tendency and dispersion
- Measures of variability
- Significance test
- Confidence limits
- Testing for outliers

Preventive Maintenance

- Schedule of maintenance tasks
- List of critical spare parts on hand

TABLE 1. (CONTINUED)

Corrective Action
Predetermined limits for data acceptability
Procedures for corrective action
Responsible individuals
Quality Assurance Reports to Management
Frequency of reporting
Responsible individuals
Significant problems and recommended solutions

1.3 Summary of Findings

Radian Corporation's field sampling and documentation procedures were highly acceptable. Additionally, it appears that the sample analyses have been performed in a responsible manner and that the reported results are most likely as good as possible considering: (1) the difficulties associated with the start-up of a new laboratory; (2) the difficult sample matrices Radian was required to work with; and (3) the methods development nature of the analyses. Furthermore, although the data quality reported by Radian (expressed in terms of percent recovery) may not be as good as hoped for in several cases, these results appear to satisfy, in principle, the objectives of the test programs.

2.0 FIELD AUDITS

2.1 Introduction

RTI performed technical systems audits at three sites where emission testing for dioxins in stack gases was underway. The locations of the test are considered confidential information by EPA, but are identified in this report as Sites ISW-A, BLB-B, and MET-A. The three emission tests, which were conducted by Radian Corporation, were similar in that they all involved the sampling of feed and process materials, and soils, in addition to the sampling of stack gases for dioxins, precursors, and HCl.

RTI's objectives during the three audits were somewhat different. For example, during the first audit RTI's primary concern was with the completion of detailed check lists covering the test procedures in use as well as those to be used for all future tests. A primary objective of the second audit was to confirm that the appropriate procedures were still in use and to observe in detail all test procedures. The final audit was intended to evaluate modifications made to Radian's continuous emission monitoring system and to the Modified Method 5 sampling trains. Although the objectives of the three audits were somewhat different, each audit included the observation of procedures and the questioning of test team personnel. Each of the three audits are described in more detail below.

2.2 Testing at Site ISW-A

On November 8, 1984, RTI performed a technical systems audit of a dioxin emission test program underway at Site ISW-A.[6] The audit was performed by Mr. Richard Crume. The goals of the audit were to: (1) evaluate Radian Corporation's adherence to the test program's test plan and QA plan; (2) document the test procedures used; and (3) make any recommendations that could improve the quality of data collected during future tests. Check lists used in evaluating Radian's procedures are presented in Appendix 8.1.

RTI's recommendations from the Site ISW-A audit are summarized in Table 2. These recommendations addressed problems which were not serious enough to invalidate the test results. However. RTI believed that implementation of the recommendations was important for two reasons: (1) the recommendations would help prevent potential problems from developing in the future: and (2) the recommendations would bring all test procedures into agreement with the written protocols. Overall, RTI was impressed with the test team and the quality of their work. Although on the day of the audit several process disruptions occurred which were beyond the control of the Radian test team, these problems were overcome without sacrificing the integrity of the test program. Thus, RTI was satisfied that the data generated by the test would be of sufficient quality to achieve the objectives of the study.

2.3 Testing at BLB-B

On February 27, 1985, RTI performed a technical systems audit of a dioxin emission test program underway at Site BLB-B.[7] The audit was performed by Mr. Richard V. Crume and Ms. Donna J. Holder. The goals of the audit were to: (1) evaluate Radian Corporation's adherence to the test program's Test Plan and QA Plan; and (2) determine whether the recommendations made by RTI as a result of the earlier audit at Site ISW-A had been fully implemented.

As with the earlier Site ISW-A audit, the RTI auditors were impressed with the Radian test team and the quality of their work. It appeared that the test team faithfully adhered to their Test Plan and QA Plan and that the required sampling procedures were carefully followed. Furthermore, with two minor exceptions (the position of the condenser on the Modified Method 5 sampling train and the marking of liquid levels on bottles), all of RTI's earlier recommendations had been implemented. The two exceptions are summarized below.

- <u>Position of Condenser on MM5 Train</u>

  The MM5 sampling train appeared to be set up and operated according to the ASME/MM5 sampling methodology specified in Radian's Test Plan, with one exception. This involved the mounting of the XAD trap condenser in a horizontal position, as opposed to the vertical orientation specified in the ASME/MM5 method. However, the hori-

TABLE 2. SUMMARY OF RECOMMENDATIONS FROM A SYSTEMS AUDIT AT SITE ISW-A

Modified Method 5 (MM5) Sampling Train

- Radian should mount the MM5 sampling train's XAD-condenser in a vertical position during future tests. Alternatively, Radian should explain in each test report: (1) why the condenser mounting position differs from the test protocol; and (2) what effect this is likely to have on the outcome of that particular series of tests (e.g., was all moisture observed to be carried forward into the resin).

Blank MM5 Sampling Train

- The front and back ends of the blank MM5 train should remain sealed throughout each test. The probe should be sealed with hexane-rinsed aluminum foil and the last impinger with a ground glass cap.

Ambient XAD Sampling Train

- Ordinarily the XAD resin trap associated with the ambient sampling train is kept in place between the 4-hour test runs and is not removed until the final test run at a given site is complete. The resin trap should be cooled between test runs as well as during the runs. This is especially important whenever the train is located in hot or variable-temperature environments.

HCl Sampling Train

- During the audit the HCl sampling probe broke, thereby invalidating the HCl results. More care should be taken in the future to avoid breakage of equipment.

Continuous Emission Monitors

- Calibration and quality control gases for the continuous emission monitors should test the entire sampling interface, beginning at the stack.

- Once the span check of a continuous monitor has been completed, the monitor's reading should be allowed to return to zero before challenging the meter with a quality control gas.

- If possible, Radian's continuous monitor data acquisition system should incorporate a time constant to average out positive and negative noise peaks in the monitor signal.

(continued)

TABLE 2. (CONTINUED)

- Temperature variations within the continuous monitor trailer should be minimized so that continuous monitor stability will be improved.
- Continuous monitor strip charts should be offset a positive 10% from zero to avoid negative drift.

Sample Handling, Transportation, and Storage

- The liquid level on all sample collection bottles should be marked at the time of collection.

Soil Sampling

- Soil sampling should be conducted over a wide area where potential dioxin contamination is most likely.
- Soil samples should be composited using an appropriate tool, such as a hexane-rinsed garden trowel.
- All debris (e.g., leaves and dead grass) should be cleaned from the ground before soil sampling is begun.

zontal mounting of the condenser was approved earlier by EPA as a modification to the ASME/MM5 methodology presented in Radian's Test Plan. (This approval was based on evidence that, for this test program, all condensed liquids are carried forward into the XAD resin regardless of whether the condenser is horizontally or vertically mounted.) Thus, RTI was satisfied that the horizontal position of the condenser was acceptable.

o <u>The Marking of Liquid Levels on Bottles</u>

In contrast to RTI's earlier recommendation, Radian continued to mark sample weights rather than liquid levels on their sample bottles. However, RTI did not consider this a serious problem, provided that Radian continued to spot-check the weights of bottles arriving in the laboratory.

Another deviation from the previous test procedures involved the solvent used for sampling train recovery, which was switched from hexane to methylene chloride. This change in solvent was recommended by EPA as a result of the high blank values which occurred during earlier tests when hexane was used as the solvent.[8] Otherwise, RTI had no objections to the procedures used by Radian. Thus, the auditors continued to believe that the Radian test team was producing acceptable data.

2.4 Testing at Site MET-A

On May 30, 1985, RTI peformed a technical systems audit of a dioxin test program underway at Site MET-A.[9] The audit was performed by Mr. Richard V. Crume and Mr. Robert S. Wright. At EPA's request, RTI's audit focused on the continuous emission monitoring system and on modifications made to the Modified Method 5 sampling train. Additionally, RTI examined other sampling systems and reviewed in-house audit data provided by Radian.

The continuous emission monitoring system was of particular interest to EPA because a new set-up, which had not been used during previous Tier 4 tests, was in use. Although the new set-up consisted of the same equipment that had previously been used, the equipment had been moved to a newly outfitted truck. RTI carefully examined the new set-up and concluded that it was satisfactory, although several minor problems were detected. RTI recommended that these problems, which are summarized below, be addressed prior to the next test program.

- O_2 Monitor. Although the O_2 monitor was operating correctly, a problem with the signal conditioning box prevented the signal from reaching the data acquisition system. Instead, 5-minute averages were taken by hand.

- SO_2 Calibration and QC Gases. The SO_2 concentrations found in the stack (about 250 ppm) were much higher than expected. As a consequence, the concentrations of the calibration gas (83.5 ppm) and QC gas (19.6 ppm) were too low to be effective. (The instrument scale was 500 ppm rather than the 100 ppm scale anticipated prior to the testing.)

- Verification of Calibration Gas. The calibration gas certifications had not been verified. Nevertheless, Radian felt that the ± 20% accuracy QA objective would cover any possible certification inaccuracy. (However, prior RTI audits of commercial "certified" calibration standards found that their certified values could be in error by greater than 20 percent. Errors of this magnitude would leave little room for other instrumental errors.)

- Calibration Gas Certifications. Several of the calibration standards had not been analyzed or re-analyzed within six months of the test. (It should be noted, however, that Radian's QA project plan does not call for periodic re-analysis of the calibration standards.)

- Calibration Standards vs. Certificates. The gas producer's calibration standards on-hand in the Radian mobile facility did not match producer's certificates on-hand.

- NO_x Monitor. The NO_x monitor drift, at ± 5 to 10%, exceeded that observed for the other monitors. Although at the time of the audit the drift was still within the acceptance limit of ± 20% for the single point response factor test, the monitor should be closely watched to prevent a worsening of drift during future tests.

The Modified Method 5 sampling train in use at the Site MET-A tests was unique in that a second XAD resin cartridge was added to the system just after the first impinger, as illustrated in Figure 1. This configuration, which was requested by EPA, was designed to mitigate concerns regarding the horizontal position of the XAD condenser. (Although the horizontal mounting of the condenser had been approved by EPA during previous Tier 4 tests, officials from a State Agency remained concerned about an increased potential for organic compounds breaking through the resin.) The set-up and operation of the second XAD resin cartridge appeared to be acceptable. Furthermore, with one exception, operation of the entire train appeared normal. The one exception involved the formation of a yellow precipitate in

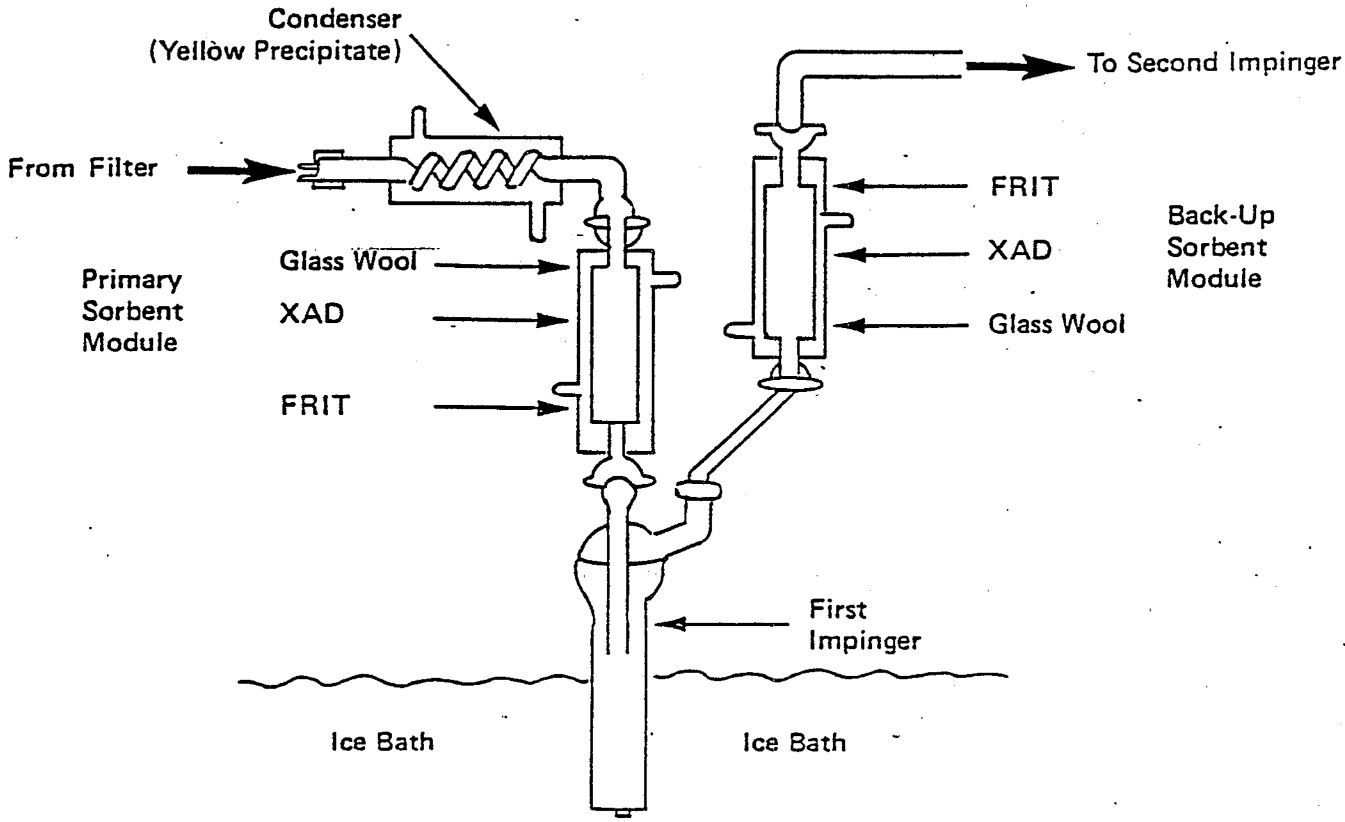

Figure 1. Configuration of primary and back-up sorbent modules in Modified Method 5 sampling train.

the condenser, between the filter and the first XAD resin cartridge. The nature of this precipitate is unknown, although its color suggests it may be chloride. Radian reported that the precipitate was easily removed with acetone.

Several other problems occurring during the test program are summarized below:

- Analytical Laboratory. Radian's mobile analytical laboratory was damaged en route to the test site and had to be left behind. Most of the laboratory equipment was salvaged and transferred to a temporary laboratory set-up at the plant. The temporary laboratory was inspected by RTI and appeared to be satisfactory.

- Filter Placement. On the first day of testing the Modified Method 5 sample train filters were placed backwards, thereby invalidating the test results. These tests were consequently repeated.

- Electrical Problems. Power supply problems forced testing to be delayed during the morning of the second day. However, these problems were solved by noon and did not seriously interfere with the test schedule.

In summary, although several problems were observed while testing was in progress, these problems did not seriously interfere with the test results. Thus, the auditors continued to be satisfied with the Radian field sampling program.

3.0 LABORATORY AUDITS

3.1 Introduction

RTI performed two technical systems audits of Radian Corporation's analytical facilities in Research Triangle Park, North Carolina. The first audit was preliminary in that the actual Tier 4 analyses had not yet been initiated. The second audit was a follow-up to the first one, and included a discussion of analytical procedures and results. The two audits are discussed below.

3.2 Initial Audit

A technical systems audit of Radian Corporation's analytical facility in Research Triangle Park, North Carolina, was conducted on June 12, 1985, by Ms. Doris Smith, Ms. Donna Holder, and Mr. Richard Crume.[10,11] The purpose of the audit was to evaluate Radian Corporation's adherence to the Tier 4 Quality Assurance Project Plan and to make any recommendations that could improve the quality of the data generated. Radian's new analytical facility was well-equipped and had recently made significant progress towards commencing sample analyses. Although many difficulties associated with a newly functioning laboratory had to be overcome, the preparation of samples for analysis had begun, and set-up procedures were underway in the mass spectrometer laboratory.

Generally, the auditors believed there was a need for the laboratory to improve its documentation procedures. For example, revisions to protocols were not adequately documented, few SOPs or protocols were available to the staff, and there were numerous blank entries in the master logbook. Furthermore, a formal plan for corrective action had yet to be implemented. Additionally, the auditors were concerned about: (1) the role of Radian's QA Officer; (2) the long storage time for XAD-2 samples; (3) the need for a dioxin sample tracking system; and (4) the use of appropriate analytical standards. RTI's concerns are summarized below.

- o Documentation procedures needed to be improved. Modifications to analytical methods and protocols should have been documented in a formal manner.
- o Protocols and SOPs should have been available to the analytical laboratory staff.
- o The QA Officer had not been actively involved in QA activities and had not demonstrated an interest in pursuing an independent, active QA role in the dioxin program.
- o The potential impact of the long storage time for the XAD-2 resin samples should have been addressed.
- o A sample tracking system, specific to the National Dioxin Study, should have been implemented within the analytical facility.
- o Standards used for calibration, quantitation, and surrogates should have been certified or traceable to certified standards.

Although the auditors identified during the audit a number of problems which should have been resolved, most of these problems were understandable given: (1) the difficulties associated with the breaking in of a new facility; and (2) the "methods development" nature of the project. Thus, the auditors were not overly concerned about the Radian analytical facilities.

3.3 Final Audit

The follow-up audit of Radian Corporation's analytical facility in Research Triangle Park, North Carolina, was conducted on September 10, 1985, by Ms. Doris Smith and Mr. Richard Crume.[12] The purpose of the follow-up audit was to determine whether Radian had satisfactorily addressed the concerns raised in the previous audit. Additionally, the auditors evaluated Radian's adherence to the Tier 4 Quality Assurance Project Plan with respect to some of the analytical procedures that could not be examined during the previous audit.

Overall, the auditors were impressed with the measures Radian had taken to satisfy the issues raised earlier. A check-list used by the auditors is presented in Table 3. Additionally, Radian's responses to the issues raised in the earlier audit are summarized below.

TABLE 3. CHECK-LIST FOR FOLLOW-UP AUDIT OF THE RADIAN/RTP ANALYTICAL FACILITY

Question	Response Yes	Response No	Comments
Will the "Level 3" report discuss the XAD "storage time" issue?	X		The XAD "storage time" issue will be discussed in the quality assurance section of the final report (i.e., the "Level 3" document).
Who currently serves as QA Officer, and what are his functions?			Mr. Mike Palazzolo currently serves as QA Officer, although he is not actually independent of project management.
What other internal QA activities are being, or have been, performed?			Mr. Ken Rozacky from Radian's Austin, Texas office has performed a thorough systems audit of the RTP analytical facilities. Additionally, he has provided three different spiked QC samples for GC/MS analysis. Mr. Mike Palazzolo routinely reviews and validates data. Ms. Joan Bursey reviews and evaluates the results from all analytical QC checks.
Have general sample preparation and analysis procedures been written down? (Can these be inspected?)	X		The sample preparation and analysis procedures have been defined and are recorded in the laboratory notebooks. Additionally, the procedures will be presented in the final report.
Will these procedures be used in processing additional samples, and will they be documented in the final report?	X		All samples are now analyzed by a standard procedure. These procedures will be discussed in the final report.

TABLE 3. (CONTINUED)

Question	Response		Comments
	Yes	No	
Has a sample tracking system specific to the National Dioxin Study been implemented within the analytical facility? (Can this be inspected?)	X		Sample identification data are entered into the master log book and then entered on a second list reserved only for dioxin program samples. The samples in the second list are categorized according to test site. Additionally, each sample has an individual data sheet which follows it throughout the laboratory.
Will the results of cross-checking and QC checks for the GC/MS system be reported in the final documentation?	X		Although these checks are performed by several different individuals, the results of the checks will be combined and discussed in the final report.
Has Radian requested certified materials from EPA/EMSL?	X		The acids have been run and the base/neutrals will be run soon. All results are ± 20 or better of the expected values. (Certified materials are not available for chlorinated biphenols.)
Is the GC/MS system functioning properly with respect to: o acceptance criteria? o tuning & calibration? o column performance? o control charts?	X		See notes below. Otherwise, the system appears to be functioning properly. Control charts and other performance data are currently in preparation for inclusion in the final report.
Are QA Objectives for precision and accuracy consistently achieved for the GC/MS analyses?		X	Some precision and accuracy (i.e., recovery) objectives have not been achieved due to the inhomogeneity of the sample matrices. Completeness objectives have been achieved.

- Protocols for sample preparation and analysis were to be included in the final report. Additionally, all procedures had been documented in laboratory notebooks. (Nevertheless, the auditors were disappointed that the procedures were still not in protocol or SOP form even though the sample preparation steps were essentially complete.)

- Although the QA Officer, Mr. Mike Palazzolo, was still not independent of line project management, he had been actively involved in QA activities and had instituted some good QA procedures. Additionally, the project had been audited internally by Radian's QA Officer from their Austin, Texas office.

- The potential impact of the long storage time for the XAD-2 resin samples was to be addressed in the final report.

- A sample tracking system, specific to the Tier 4 sampling activities, had been implemented.

- Some cross-checking of analytical quantitation standards with certified materials had taken place, and additional cross-checking was planned.

In summary, the auditors found that Radian had essentially responded to each of the earlier QA concerns in a positive and acceptable manner.

3.4 Overall Assessment

Radian Corporation's interim report of Tier 4 analytical results indicates that the data quality objectives for the analyses of chlorinated phenols, chlorobenzenes, and polychlorinated biphenyls, as expressed in Radian's quality assurance plan, may not be achieved for all compounds.[13] (The quality assurance plan establishes data quality objectives of $\pm$ 50% for both precision and accuracy.) However, the interim report makes several important arguments regarding the difficulty of these analyses. For example:

- "There are no directly comparable values for surrogate recoveries available from the chemical literature for most of the feed material."

- "There are no published procedures for sample preparation for most of the media analyzed."

- "Due to [a variety of] sample matrix problems, a certain degree of method development was necessary; however, no resources were alloted for optimization studies to improve method performance in terms of absolute percent surrogate recovery and precision."

- "[Since large sample sizes (10 to 50g) were taken,] even the low recovery of surrogates gives a good estimate of the level of and/or actual presence of the target compounds. For example, a 10 percent surrogate recovery results in a 20-100 ppb sensitivity for the target compounds in a 50g sample."

- "With consideration for the sensitivity of the analytical techniques, a sample size was selected for extraction/purification so that, even if the recovery of the target compound were as low as 10-30%, a level of analyte well above instrumental detection limits would be delivered to the analytical system. Therefore, detection limits for the method in a 50g sample for 10-30% recovery would be 20-100 ppb."

In addition to the above comments, the interim report presents several justifications (e.g., high volatility, low purity, limited solubility, and high molecules weight) for the poor recoveries associated with several surrogate compounds. The other surrogate compounds had significantly better recoveries.

In consideration of the above comments as well as the outcomes of the two laboratory audits, it appears that Radian's analyses have been performed in a responsible manner and that the reported results are most likely as good as possible under the circumstances. Furthermore, although the data quality reported by Radian (expressed in terms of percent recovery) may not be as good as hoped for in several cases, the results appear to satisfy, in principle, the objectives of the test program. (That is, the objectives, although quantitative, allow for a large margin of error, as evidenced by the established data quality objectives for precision and accuracy of $\pm$ 50%.)

4.0 PERFORMANCE AUDITS

4.1 Introduction

RTI's performance evaluation audits were intended to evaluate the following measurement and analytical systems: (1) dry gas meters; (2) computerized data acquisition and associated calculations; (3) fuel oil analyses for chlorine; (4) HCl train analyses; and (5) dioxin analyses by the Troika Laboratories. Most of these evaluations were initiated simultaneously with the first two technical systems audits. The results of RTI's performance evaluation audits are discussed below.

4.2 Dry Gas Meters

Three sample train meter boxes were in use during Radian's Site ISW-A tests. The calibration of these meter boxes was evaluated by providing Radian with a calibrated orifice.[14,15] Radian was given all the information needed to conduct the audit themselves, except that the orifice calibration factor was not provided. Thus, Radian had no clue as to what orifice flow rates were being measured by the meter boxes. Radian returned the orifice data to RTI for evaluation. The calibrated orifice used in the audit was obtained from EPA's Environmental Monitoring Systems Laboratory in Research Triangle Park, North Carolina.

The results of the calibrated orifice audit are presented in Table 4. These data indicate that the meter box readings and the actual flow through the calibrated orifice agreed very well. (Agreement within ± 5% is considered good.) The values presented in Table 4 were calculated by RTI, based on the raw data provided by Radian and on the orifice calibration factor provided by EPA.

During the technical systems audit of the Site BLB-B tests, the above procedure was repeated. The results were also quite good, as shown in Table 5.

TABLE 4. COMPARISON OF METER BOX AND CALIBRATED ORIFICE VOLUMES FOR THE SITE ISW-A TESTS

Meter Box I.D.	Final Meter Volume (ft^3)	Final Orifice Volume (ft^3)	Difference (%)
3	10.327	10.517	- 1.8
5	10.597	10.517	+ 0.8
7	10.609	10.517	+ 0.9

TABLE 5. COMPARISON OF METER BOX AND CALIBRATED ORIFICE VOLUMES FOR THE SITE BLB-B TESTS

Meter Box I.D.	Final Meter Volume (ft^3)	Final Orifice Volume (ft^3)	Difference (%)
RAC #4	10.252	10.525	- 2.6
RAC #6	10.533	10.495	0.4
RAC #9	10.178	10.545	- 3.5

4.3 Computer Systems

During the Site ISW-A tests, Radian's computerized calculations were evaluated by providing Radian with a hypothetical set of data for which expected calculated results were already known by RTI.[14] Radian's results would be expected to agree with RTI's previously calculated results, unless Radian's calculations were in error. As shown in Table 6, Radian's results are almost exactly the same as the expected values. Thus, it appears that Radian's computer program was correctly programmed.

4.4 Fuel Oil Analyses for Chlorine

During the Site ISW-A tests RTI provided Radian with two audit samples consisting of No. 2 fuel oil spiked with a known quantity of methylene chloride.[16] These samples were prepared by staff members of RTI's Environmental Chemistry Department. Two chlorine concentration levels were prepared: a high value (4432 ppm) and a low value (443 ppm). RTI's selection of the high and low chlorine concentration levels was based on our concern that the analytical method to be used (ASTM Method D-808) has a relatively high detection limit of about 1000 ppm, and that concentrations lower than 1000 ppm may be encountered during the test program.

RTI verified the expected concentrations of its audit samples using the preferred Modified ASTM D-808 Method (i.e., Parr bomb combustion, Na_2CO_3 rinse, and ion chromatography analysis). The percent recoveries using this method ranged from about 60 to 80%, as expected. The calculation of these recoveries took into account the "blank" (i.e., unspiked fuel oil) value of about 55 ppm of total chlorine. The expected and reported results for the spiked fuel oil analyses are presented below.

Expected Value	Value Reported By Radian	Percent Difference[a]
4432 ppm	0.48% (approx. 4800 ppm)	+9.1%
443 ppm	0.12% (approx. 1200 ppm)	+170%

[a] Calculations based on two significant figures.

TABLE 6. COMPARISON OF SOURCE TEST DATA CALCULATIONS BY RADIAN CORPORATION AND RTI

Calculation	Radian Value[a]	RTI Value
Dry gas volume at standard conditions	13.842 dscf	13.842 dscf
Water vapor volume at standard conditions	12.237 scf	12.237 scf
Stack gas moisture	46.923%	46.923%
Dry stack gas mole fraction	0.53077	0.53077
Dry stack gas molecular weight	29.478	29.478
Wet stack gas molecular weight	24.092	24.092
Stack gas velocity at actual conditions	1106.851 fpm[b]	1106.851 fpm
Actual stack gas volumetric flow rate	730.522 acfm	730.522 acfm
Dry stack gas volumetric flow rate, standard conditions	252.117 dscfm	252.117 dscfm
Isokinetic sampling rate	132.952%	132.759%
Particulate concentration	0.0038397 g/acf	0.0038398 g/acf
Particulate emission rate	0.3710983 lb/hr	0.3710395 lb/hr

[a] Radian presents the results of its calculations in both English and Metric units. The English/Metric conversion factor for each calculation was examined, and in each case the conversion factor used was correct.

[b] Radian correctly uses a pitot constant of 0.83 in this calculation. However, Radian's computer printout erroneously shows a pitot constant of 0.84 in the calculation. The reason for this is not clear.

The data presented above indicate that the analytical method used to measure total chlorine levels in fuel oil is inaccurate at moderate to low chlorine concentration levels.

As a result of the above data, the chlorine-in-fuel oil audit was repeated using a second set of audit samples.[17] Better results on the second set of audit samples were expected, since Radian had indicated that the more desirable method of analysis would be implemented. However, as indicated below, the analytical results for the second set of samples were still not acceptable.

Expected Value	Value Reported By Radian
4432 ppm	BDL[a]
4432 ppm[b]	BDL
433 ppm	BDL
433 ppm[b]	BDL

[a] BDL = Below Detection Limit.
[b] Duplicate sample.

Note that the second set of samples are identical to the first set (i.e., from the same batch), except that the second set was submitted in duplicate.

Due to Radian's continuing concern about the quality of the analyses, the samples were given to a subcontractor, Research Triangle Institute, to analyze. Radian provided its own QA samples for these analyses.

4.5 HCl Train Analyses

Two chloride ion audit samples were submitted to Radian for analysis by ion chromatography.[17] The samples consisted of actual impinger water taken from an HCl sampling train used in another test program. The results of the analyses are presented below:

Expected Value	Value Reported By Radian	Percent Difference
27.6 mg/l	27 mg/l	-3.6%[a]
8.1 mg/l	9 mg/l	13%[b]

[a] Calculation based on two significant figures.
[b] Calculation based on one significant figure.

RTI considers these results to be acceptable, although note that one reported value exceeds Radian's data quality objective for the HCl train of ± 7%. (Radian's 13% error may actually be less significant, since there is a margin of error associated with RTI's "expected concentration" of roughly ± 3%.)

4.6 Dioxin Analyses by Troika

The Troika Laboratories were given four 2378-TCDD audit samples for analysis. Two of the samples, "Urban Particulate" and "Urban Dust," were obtained from the National Bureau of Standards and were verified by DOW Chemical.[18,19] The two other samples, both 2378-TCDD in isooctane, were obtained from and verified by the National Bureau of Standards.[20,21] The results of Troika's analyses of these audit samples are excellent, as indicated in Table 7.

TABLE 7. EXPECTED AND REPORTED CONCENTRATIONS OF 2378-TCDD IN AUDIT SAMPLES

Audit Sample Description	Radian I.D.	Expected Value	Value Reported By Troika
Dioxin in NBS Urban Particulate (SRM 1648)	AW-68 02-HLD DQ000218	47 ppt (0.05 ppb)	0.07 ppb
Dioxin in NBS Urban Dust (SRM 1649)	AW-67 02-LLD DQ000217	6.7 ppt (0.007 ppb)	Not Detected[a]
NBS Dioxin in Isoctane	DT-109 TCDD-AUDIT A DQ005633	98.3 ppb (67.8 ng/ml)[b]	68 pg/µl (68 ng/ml)
Same	DT-110 TCDD-AUDIT B DQ005634	98.3 ppb (67.8 ng/ml)[b]	69 pg/µl (69 ng/ml)

[a] The detection limit for this method was reported by Troika as 0.04 ppb (40 ppt).

[b] This value is reported by NBS at 23°C.

5.0 REVIEWS OF QUALITY ASSURANCE PROJECT PLANS AND SITE-SPECIFIC TEST PLANS

As part of RTI's support of the Tier 4 sampling and analytical activities, two drafts and a final version of Radian's Quality Assurance Project Plan were reviewed.[22,23,24] Overall, the quality of the reports was good. The final report, in particular, reflected a strong effort by Radian to present the latest information and procedures, based on the experience gained over the first half-year of testing. For example, the final report was updated to include the following information which was absent from the earlier drafts:

- QA objectives for total chlorine.
- Rationale for horizontal mounting of MM5 condenser coil.
- Use of methylene chloride in MM5 rinsing and recovery operations.
- Addition of MM5 "proof" blank to check effectiveness of glassware cleaning procedures.
- Use of either NaOH or KOH in first two HCl train impingers.
- Addition of flow chart for precursor sample preparation and pre-cleaning.
- Inclusion of section on fuel oil chlorine analysis.
- Inclusion of equations for calculating flue gas HCl concentrations for black liquor recovery boilers.
- Addition of MM5 audit checklist.
- Revised schedule of project activities.

In addition to the above information, Radian presented in the final report acceptable responses to most of the issues raised by RTI during earlier reviews. These issues and responses are presented in Table 8. (However, RTI recommends that several of the issues still not satisfactorily

TABLE 8. COMMENTS AND RESPONSES FOR THE FINAL VERSION OF RADIAN'S QA PROJECT PLAN

RTI Comment on Previous Draft	Radian Responded to Comment?		Discussion
	Yes	No	
I. Analytical Procedures			
MS pressure appears to be unusually high	X		The reference to MS pressure has been eliminated from the text.
Add discussion of problems associated with derivatization		X	Little additional information has been added regarding: (1) completeness of conversion; and (2) differentiation of derivatization rate of chlorine
Use Ballschmiter numbers	X		
List CP isomers	X		
Clarify acceptance criteria for PCB analyses		X	Tables 3-1 and 9-1 still appear to present different acceptance criteria.
Present criteria for evaluating column performance	X		
Clarify use of surrogates and internal standards	X		The text now distinguishes between internal standards and surrogates, and their time of use.
Explain whether internal or external standards will be used for calibration		X	Section 9.2.2.1 continues to refer to the use of external standards for calibration, whereas Section 7.2.2.2 specifies that internal standards will be used.

TABLE 8. (CONTINUED)

RTI Comment on Previous Draft	Radian Responded to Comment?		Discussion
	Yes	No	
II. <u>Quality Assurance Objectives</u>			
Correct "percent difference" equation	X		
The accuracy objective for "total chlorine" appears to be too large	X		This objective has been revised from ± 50% to ± 30%.
III. <u>Internal Quality Control Checks</u>			
Table 9-4 presents the incorrect equation for "percent recovery"		X	The formula presented in Table 9-4 for "percent recovery" actually calculates instead the "percent difference" between the expected and measured surrogate concentrations.
IV. <u>Modified Method 5 Procedures</u>			
The latest approved draft of the ASME procedure should be used	X		This procedure has been added to the QA Plan.
The procedure should be indentified as an ASME procedure; pages should be numbered	X		

responded to in Table 8 be addressed prior to the completion of Radian's final report. Also, several errors in Section 8 of the report regarding Equations 8-18, 8-19, and 8-21 need to be corrected.)

In addition to the QA Project Plans, RTI reviewed four draft Radian Site-Specific Test Plans and one draft Midwest Research Institute (MRI) Work/QA Plan.[25,26,27,28,29] (The MRI Plan involved dioxin testing performed for EPA's Mr. Robert Kramer.) RTI considered these Plans acceptable, provided that the review comments were addressed in the final versions. These comments covered a wide variety of issues, including: (1) representative incinerator operation; (2) fuel oil chlorine analyses; (3) soil sampling procedures; (4) combustion air sampling; (5) data assessment; (6) analytical methodology; and (7) overall QA/QC considerations.

6.0 SUMMARY AND CONCLUSIONS

Research Triangle Institute's (RTI's) quality assurance support for Tier 4 (Combustion Sources) of the National Dioxin's Study included the following activities:

- Field Audits: Systems audits were conducted at three Radian Corporation test sites.
- Laboratory Audits: Initial and follow-up systems audits were conducted at Radian's Research Triangle Park analytical facilities.
- Performance Audits: The following measurement systems were evaluated using performance audits:

  A. Dry gas meters,
  B. Computer systems,
  C. Fuel oil analyses for chlorine,
  D. HCl train analyses,
  E. Dioxin analyses by Troika.

- Reviews of Quality Assurance Project Plans and Site-Specific Test Plans.

Overall, RTI was satisfied with the outcome of the above audit and document review activites. In particular, the field sampling activities appeared to be well executed and proceeded smoothly, and the required documentation (i.e., quality assurance and test plans) was well written. When significant problems did occur (e.g., with the effectiveness of the sampling train solvent rinse, with the chlorine-in-fuel oil analyses, and with certain laboratory procedures), Radian was able to find acceptable solutions without sacrificing data quality. Although Radian's analytical operations did not proceed as smoothly as desired, the problems encountered are understandable in light of: (1) the difficulties associated with the start-up of a new laboratory; (2) the difficult sample matrices Radian was required to work with; and (3) the methods development nature of the analyses.

In summary, Radian's field sampling and documentation procedures were highly acceptable. Additionally, it appears that the sample analyses have been performed in a responsible manner and that the reported results are most likely as good as possible under the circumstances. Furthermore, although the data quality reported by Radian (expressed in terms of percent recovery) may not be as good as hoped for in several cases, these results appear to satisfy, in principle, the objectives of the test program.

7.0 REFERENCES

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8.0 APPENDIX

8.1 Check Lists from Audit of Testing at Site ISW-A

METHOD 5 SAMPLING CHECK LIST*

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Calibration of dry gas meter against standard	X		Footnote A
Dry gas meter reading within ± 5% of calibration orifice			Footnote B
Calibration of stack temperature sensor against reference thermometer	X		
Nozzle calibrated to nearest 0.025 mm (0.001 in); size properly selected	X		
Trains correctly set-up and leak-checked (<4% or 0.00057 m^3/min, 0.02 ft^3/min, whichever is less); see attached figure.	X		
Train components are clean and free of breaks, cracks, and leaks; probe liner is clean and leak free at 380 mm (15 in) Hg	X		
Pitot tube lines are free of plugs or leaks	X		
Probe heating system is operating	X		
Pitot tube and temperature sensor are properly attached to probe	X		
Pitot tubes, meter box, and temperature sensors are recently calibrated	X		Footnote C

*This check list was used to evaluate the Modified Method 5 sampling train.

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
Nozzle and pitot tube are parallel to stack wall	X		
Filter holder temperature remains 120 ±14°C (248 ± 25°F) throughout test	X		
Sample gas temperature at last impinger remains <20°C (68°F) throughout test	X		
Isokinetic sampling within ±10%; checked every five minutes	X		
Sample site selection is appropriate distance downstream from flow disturbance	X		Footnote D
Filters are free of irregularities, properly installed, and properly labeled	X		
Operators have access to test protocol and methods; data sheets available; equipment in good repair	X		Footnote E
All data recorded; calculations checked; at least one decimal point beyond that of acquired data is retained	X		Footnote F
All impingers remain cooled in an ice bath at all times	X		
Full stack traversing being conducted	X		Footnote G

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	NO	COMMENTS
Sample recovery:			
Method prevents contamination	X		
Graduated cylinder subdivisions are < 2 ml	X		
Cap placed over nozzle tip	X		
All openings capped during train disassembly	X		Footnote H
Clean-up area clean and protected from wind	X		Footnote I
No particulate spilled	X		
No visible particles remain inside probe	X		
Sample containers labeled, sealed tight, liquid level marked		X	Footnote J

MODIFIED METHOD 5 ADDENDUM

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
Cartridge (with XAD) mounted vertically atop the first impinger		X	Footnote K
Cyclone, flask, and filter holder maintained (thermostatically) at a temperature of 120°C ± 14°C (248°F ± 25°F)	X		
Sample gas and XAD cooled to ≤20°C	X		Footnote L
XAD resin never subject to temperature greater than 100°C during preparation, transfer, and storage	X		
Greaseless types of joints and seals used in train	X		
XAD storage under refrigeration in either air tight glass containers or under ethanol or methanol	X		
Storage time of XAD after cleaning ≤ 2 months	X		
Samples of XAD batch analyzed just prior to use in the field	X		
Sample recovery			
XAD-2 handling procedure properly implemented	X		
Same solvents used for resin extraction and cleaning sequence	X		

FOOTNOTES--MODIFIED METHOD 5 SAMPLING CHECKLIST

A The dry gas meter was calibrated against a wet test meter standard several weeks prior to the audit.

B Radian will check the calibration of the dry gas meter using a calibrated orifice supplied by RTI. The calibration data will be returned to RTI for analysis. (Radian was not provided with the orifice calibration factor.)

C All calibrations were performed about 4 weeks prior to the audit.

D The sampling location was about 13 feet from the ambient air intake damper.

E The sampling equipment was rather old, but appeared to be well maintained.

F Audit data were used to examine the accuracy of Radian's computerized calculations.

G Twenty-four point traverses were performed.

H Openings capped using hexane-rinsed aluminum foil.

I The cleanup area was located inside a closed trailer.

J Liquid levels were not marked on bottles. Instead, bottle weights were taken both before and after the bottles were filled, and this information was recorded on the bottle labels.

K The XAD cartridge was vertical, but the condenser was mounted in a horizontal position.

L Cooling was achieved using water from the impinger ice bath.

METHOD 5 SAMPLING CHECK LIST*

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	NO	COMMENTS
Calibration of dry gas meter against standard			Footnote A
Dry gas meter reading within ± 5% of calibration orifice			NA
Calibration of stack temperature sensor against reference thermometer			NA
Nozzle calibrated to nearest 0.025 mm (0.001 in); size properly selected			NA
Trains correctly set-up and leak-checked (<4% or 0.00057 m^3/min, 0.02 ft^3/min, whichever is less); see attached figure.			NA
Train components are clean and free of breaks, cracks, and leaks; probe liner is clean and leak free at 380 mm (15 in) Hg			NA
Pitot tube lines are free of plugs or leaks			NA
Probe heating system is operating			NA
Pitot tube and temperature sensor are properly attached to probe		X	
Pitot tubes, meter box, and temperature sensors are recently calibrated			NA

*This check list was used to evaluate the Blank Modified Method 5 train.
NA = Not Applicable

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	NO	COMMENTS
Nozzle and pitot tube are parallel to stack wall			NA
Filter holder temperature remains 120 ±14°C (248 ± 25°F) throughout test			NA
Sample gas temperature at last impinger remains <20°C (68°F) throughout test			NA
Isokinetic sampling within ±10%; checked every five minutes			NA
Sample site selection is appropriate distance downstream from flow disturbance			Footnote B
Filters are free of irregularities, properly installed, and properly labeled	X		
Operators have access to test protocol and methods; data sheets available; equipment in good repair	X		
All data recorded; calculations checked; at least one decimal point beyond that of acquired data is retained			NA
All impingers remain cooled in an ice bath at all times		X	
Full stack traversing being conducted			NA

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
Sample recovery:			
Method prevents contamination			NA
Graduated cylinder subdivisions are < 2 ml			NA
Cap placed over nozzle tip		X	Footnote C
All openings capped during train disassembly	X		
Clean-up area clean and protected from wind	X		
No particulate spilled			NA
No visible particles remain inside probe			NA
Sample containers labeled, sealed tight, liquid level marked	X		

MODIFIED METHOD 5 ADDENDUM

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Cartridge (with XAD) mounted vertically atop the first impinger		X	Footnote D
Cyclone, flask, and filter holder maintained (thermostatically) at a temperature of 120°C ± 14°C (248°F ± 25°F)			NA
Sample gas and XAD cooled to ≤20°C			NA
XAD resin never subject to temperature greater than 100°C during preparation, transfer, and storage	X		
Greaseless types of joints and seals used in train	X		
XAD storage under refrigeration in either air tight glass containers or under ethanol or methanol	X		
Storage time of XAD after cleaning ≤ 2 months	X		
Samples of XAD batch analyzed just prior to use in the field	X		
Sample recovery	X		
XAD-2 handling procedure properly implemented	X		
Same solvents used for resin extraction and cleaning sequence	X		

FOOTNOTES--BLANK MODIFIED METHOD 5 SAMPLING CHECKLIST

A The blank train did not incorporate either a meter box or a probe nozzle. The train consisted only of the probe, filter, condenser, resin, and impingers. The impingers were left empty, and the impinger box was not filled with ice water.

B The train was located on the roof of the plant, at the base of the stack scaffolding. The distance from the train to the top of the two incinerator stacks was about 30 feet.

C Both ends of the train were left unsealed while testing was in progress.

D The XAD cartridge was vertical, but the condenser was mounted in a horizontal position.

METHOD 5 SAMPLING CHECK LIST*

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Calibration of dry gas meter against standard	X		Footnote A
Dry gas meter reading within ± 5% of calibration orifice			Footnote B
Calibration of stack temperature sensor against reference thermometer			NA
Nozzle calibrated to nearest 0.025 mm (0.001 in); size properly selected			NA
Trains correctly set-up and leak-checked (<4% or 0.00057 m^3/min, 0.02 ft^3/min, whichever is less); see attached figure.	X		
Train components are clean and free of breaks, cracks, and leaks; probe liner is clean and leak free at 380 mm (15 in) Hg	X		
Pitot tube lines are free of plugs or leaks			NA
Probe heating system is operating			NA
Pitot tube and temperature sensor are properly attached to probe			NA
Pitot tubes, meter box, and temperature sensors are recently calibrated			NA

*This check list was used to evaluate the Ambient Modified Method 5 Train.
NA = Not Applicable

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Nozzle and pitot tube are parallel to stack wall			NA
Filter holder temperature remains 120 ±14°C (248 ± 25°F) throughout test			NA
Sample gas temperature at last impinger remains <20°C (68°F) throughout test			NA
Isokinetic sampling within ±10%; checked every five minutes			NA
Sample site selection is appropriate distance downstream from flow disturbance			Footnote C
Filters are free of irregularities, properly installed, and properly labeled			NA
Operators have access to test protocol and methods; data sheets available; equipment in good repair	X		Footnote D
All data recorded; calculations checked; at least one decimal point beyond that of acquired data is retained	X		Footnote E
All impingers remain cooled in an ice bath at all times			NA
Full stack traversing being conducted			NA

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Sample recovery:			
Method prevents contamination	X		
Graduated cylinder subdivisions are < 2 ml	X		
Cap placed over nozzle tip	X		
All openings capped during train disassembly	X		Footnote F
Clean-up area clean and protected from wind	X		Footnote G
No particulate spilled			NA
No visible particles remain inside probe			NA
Sample containers labeled, sealed tight, liquid level marked		X	Footnote H

MODIFIED METHOD 5 ADDENDUM

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
Cartridge (with XAD) mounted vertically atop the first impinger	X		
Cyclone, flask, and filter holder maintained (thermostatically) at a temperature of 120°C ± 14°C (248°F ± 25°F)			NA
Sample gas and XAD cooled to ≤20°C			Footnote I
XAD resin never subject to temperature greater than 100°C during preparation, transfer, and storage	X		
Greaseless types of joints and seals used in train	X		
XAD storage under refrigeration in either air tight glass containers or under ethanol or methanol	X		
Storage time of XAD after cleaning ≤ 2 months	X		
Samples of XAD batch analyzed just prior to use in the field	X		
Sample recovery			
XAD-2 handling procedure properly implemented	X		
Same solvents used for resin extraction and cleaning sequence	X		

FOOTNOTES--AMBIENT MODIFIED METHOD 5 SAMPLING CHECKLIST

A The dry gas meter was calibrated against a wet test meter standard several weeks prior to the audit.

B Radian will check the calibration of the dry gas meter using a calibrated orifice supplied by RTI. The calibration data will be returned to RTI for analysis. (Radian was not provided with the orifice calibration factor.)

C The train was located on the roof of the plant at the base of the stack scaffolding. The distance from the train to the top of the two incinerator stacks was about 30 feet.

D The sampling equipment was rather old, but appeared to be well maintained.

E Audit data were used to examine the accuracy of Radian's computerized calculations.

F Openings capped using hexane-rinsed aluminum foil.

G The cleanup area was located inside a closed trailer.

H Liquid levels were not marked on bottles. Instead, bottle weights were taken both before and after the bottles were filled, and this information was recorded on the bottle labels.

I Since the resin is cooled with ambient air between tests, a temperature of less than 20 °C may be difficult to maintain on a warm day.

METHOD 5 SAMPLING CHECK LIST *

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Calibration of dry gas meter against standard	X		Footnote A
Dry gas meter reading within ± 5% of calibration orifice			Footnote B
Calibration of stack temperature sensor against reference thermometer	X		
Nozzle calibrated to nearest 0.025 mm (0.001 in); size properly selected	X		
Trains correctly set-up and leak-checked (<4% or 0.00057 m^3/min, 0.02 ft^3/min, whichever is less); see attached figure.	X		
Train components are clean and free of breaks, cracks, and leaks; probe liner is clean and leak free at 380 mm (15 in) Hg		X	Footnote C
Pitot tube lines are free of plugs or leaks	X		
Probe heating system is operating	X		
Pitot tube and temperature sensor are properly attached to probe	X		
Pitot tubes, meter box, and temperature sensors are recently calibrated	X		Footnote D

* This check list was used to evaluate the HCl sampling train.

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
Nozzle and pitot tube are parallel to stack wall	X		
Filter holder temperature remains 120 ±14°C (248 ± 25°F) throughout test	X		
Sample gas temperature at last impinger remains <20°C (68°F) throughout test	X		
Isokinetic sampling within ±10%; checked every five minutes	X		
Sample site selection is appropriate distance downstream from flow disturbance	X		Footnote E
Filters are free of irregularities, properly installed, and properly labeled	X		
Operators have access to test protocol and methods; data sheets available; equipment in good repair	X		Footnote F
All data recorded; calculations checked; at least one decimal point beyond that of acquired data is retained	X		Footnote G
All impingers remain cooled in an ice bath at all times	X		
Full stack traversing being conducted		X	Footnote H

METHOD 5 SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Sample recovery:			
Method prevents contamination	X		
Graduated cylinder subdivisions are < 2 ml	X		
Cap placed over nozzle tip	X		
All openings capped during train disassembly	X		Footnote I
Clean-up area clean and protected from wind	X		Footnote J
No particulate spilled	X		
No visible particles remain inside probe	X		
Sample containers labeled, sealed tight, liquid level marked			Footnote K

FOOTNOTES--HCl SAMPLING CHECKLIST

A The dry gas meter was calibrated against a wet test meter standard several weeks prior to the audit.

B Radian will check the calibration of the dry gas meter using a calibrated orifice supplied by RTI. The calibration data will be returned to RTI for analysis. (Radian was not provided with the orifice calibration factor.)

C At the end of the run on the day that the audit took place, the test team discovered that the HCl sampling probe had cracked. Since the time at which the crack occurred could not be determined, the run was invalidated.

D All calibrations were performed about 4 weeks prior to the audit.

E The sampling location was about 13 feet from the ambient air intake damper.

F The sampling equipment was rather old, but appeared to be well maintained.

G Audit data were used to examine the accuracy of Radian's computerized calculations.

H Sampling was performed at the point of average velocity in the stack, as specified in the Radian test plan.

I Openings capped using hexane-rinsed aluminum foil.

J The cleanup area was located inside a closed trailer.

K Liquid levels were not marked on bottles. Instead, bottle weights were taken both before and after the bottles were filled, and this information was recorded on the bottle labels.

CONTINUOUS EMISSION MONITOR CHECK LIST

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
I. Gases Monitored (Instrument to be Used):			
CO_2 (Anarad 412)	X		
CO (Anarad 412)	X		
NO_2 (Teco 10)	X		
NO (Teco 10)	X		
SO_2 ( )			
O_2 (Beckman 755)	X		
THC (Beckman 402)	X		
Opacity ( )			
Other ( )			
II. Calibration: Performed at least every 24 hours by experienced personnel following manufacturer's instructions	X		Footnote A
Cal gases verified within last 6 months; tank pressure above 100 psi	X		Footnote B
Calibration checks entire sampling interface	X		Footnote C
Zero and span values checked at the beginning and end of each run	X		
III. Records: Data continuously recorded on strip-charts or computer, and periodically averaged	X		Footnote D

CONTINUOUS EMISSION MONITOR CHECK LIST (continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	NO	COMMENTS
III. Con't			
Strip-chart zero offset 10% at recorder; charts clearly identified	X		Footnote E
All calibrations, maintenance, and problems recorded in log book	X		
Unusual readings are immediately brought to the attention of the team leader	X		
IV. General:			
Instrument/stack interface appears adequate:			
In-stack sample probe	X		
Coarse filter	X		
Heat-traced sample line	X		
Sampling pump	X		
Moisture removal	X		
Fine filter	X		
Manifold design	X		
Drift appears normal	X		Footnote F
Representative sampling site selected	X		
Maintenance procedure followed	X		

FOOTNOTES--CONTINUOUS EMISSION MONITOR SAMPLING CHECKLIST

A A three-point calibration was performed at the beginning of the test program. Two-point calibrations are performed at the beginning and end of each test run.

B Quality control gas cylinders are certified by the vendor against an NBS standard. All gases are periodically returned to the vendor for recertification. Pressures are not allowed to go below 200 psi.

C The calibration gases check the entire interface except for the sample line leading from the stack sampling probe.

D A data acquisition system records data at 5-minute intervals. This system does not average readings over time. Instead, it takes an instantaneous reading without regard for whether that reading is at the top, center, or bottom of a noise fluctuation.

E The offset is 5 percent.

F All data are corrected for drift at the end of each run. The NO_x monitor, which tends to be more temperature-sensitive than the other instruments, exhibited a greater drift than the other instruments. This drift, however, was not excessive.

PROCESS SAMPLE CHECK LIST

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Type of sample collected: ASH: Incinerator bottom ash FUEL: No. 2 Fuel oil FEED: Waste Feed QUENCH WATER: ______ OTHER: ______	 X X X		Footnote A
Collection point: Incinerator			
Sample containers are carefully sealed; liquid levels marked		X	Footnote B
Samples are representative in terms of: Process variations with time The total quantity of material under investigation	X		Footnote C
Samples are composited and/or homogenized, where appropriate	X		
A sufficient quantity of sample is collected	X		
Amber glassware is used for all liquid sample collection	X		
All glassware sealed with aluminum foil or Teflon-lined cap	X		

PROCESS SAMPLE CHECK LIST

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
All sample containers are solvent cleaned prior to reuse; solvent will not interfere with analytical method	X		
Sample containers are clearly labeled	X		
All samples are properly preserved, if not extracted within established time limit	X		

FOOTNOTES--PROCESS SAMPLE CHECKLIST

A The waste feed consisted of paint sludge, wood and plastic cutoffs, crate parts, paper, and cardboard.

B Liquid levels were not marked on bottles. Instead, bottle weights were taken both before and after the bottles were filled, and this information was recorded on the bottle labels.

C Representative waste feed samples were difficult to obtain due to the bulkiness of some materials and the thick consistency of the paint sludge. The test crew did the best they could under the circumstances.

SAMPLE HANDLING, TRANSPORTATION, AND STORAGE CHECK-LIST

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
All potentially hazardous samples are handled according to established safety procedures	X		
All sample containers are tightly sealed and carefully packaged for shipment. Plastic or aluminum wraps are used where appropriate	X		Footnote A
Samples are not stored or packaged with potential contaminants (In some cases, audit samples may be a source of contamination for other samples)	X		
Precautions should be taken in the field to avoid contamination (e.g., work areas should be clean; airborne dust should be avoided; workers should wear clean, disposal gloves)	X		
A sample custody log and a record of all field activities should be maintained; custody sheets should accompany samples during shipments	X		
Samples should be carefully labeled	X		
The duration of sample transportation and storage should not exceed the maximum recommended holding time for the sample	X		
Samples should be stored at appropriate temperatures	X		
Changes in ambient temperature and pressure during shipment (e.g., by air) should be considered	X		

FOOTNOTES--SAMPLE HANDLING, TRANSPORTATION, AND STORAGE CHECKLIST

A During a preceding field test (the second test site of this study), several bottles broke during shipment. To avoid this problem in future tests, bottles are now sealed inside two insulated plastic bags, separated by packing material. Under these circumstances future breakage seems unlikely.

SOIL SAMPLING CHECK LIST

REQUIREMENT OR PROCEDURE	ACHIEVED?		COMMENTS
	YES	NO	
The sampling technique selected is appropriate given the QA objectives of the study	X		
The sample location, sample depth, and number of samples collected are appropriate for the study	X		Footnote A
Sample segmenting or homogenization procedures used where necessary			Footnote B
Any deviations from the sampling QA plan are documented and justified	X		
Debris properly cleared from sampling location			Footnote C
All equipment thoroughly cleaned before taken to the field; any solvent rinse used will not interfere with the sample analysis	X		
All cleaned scoops, spoons, coring tubes, augers, etc. should be wrapped in aluminum foil during storage and transfer	X		
Separate onsite area designated for cleaning and decontamination of sampling equipment, if required	X		
Proper container selection; wide-mouth amber glass jars preferred	X		

SOIL SAMPLING CHECK LIST (Continued)

REQUIREMENT OR PROCEDURE	ACHIEVED? YES	ACHIEVED? NO	COMMENTS
Samples stored under appropriate conditions (e.g., darkness or refrigeration)	X		
All jars have screw-type lids lined with a material that will not cause interference with the analytical method or react with any compounds in the soil	X		
Plastic bag secured around sample containers to avoid contamination	X		
Appropriate labeling and chain of custody procedures followed for all containers	X		
All pertinent information recorded in log book	X		Footnote D

FOOTNOTES--SOIL SAMPLING CHECKLIST

A Five samples were collected at 2-foot intervals in each of two rows. The rows were parallel and were spaced about 3 feet apart, as illustrated below.

X^1 X^3 X^5 X^7 X^9

X^2 X^4 X^6 X^8 X^{10}

(The samples are numbered according to the order in which they were collected.)

Each sample was collected by: (1) forcing the bulb planter about 3 inches into the ground; (2) emptying the contents into a bucket; (3) forcing the planter into the ground a second time at an adjacent location; and (4) again emptying the contents into the bucket~~. Once~~ two samples (i.e., four bulb planters) were collected, the bucket was returned to the laboratory area where the contents of the bucket was mixed, and a portion transferred to an amber glass bottle. Transfers were made by a member of the test crew wearing a plastic glove. The remainder of the bucket contents was discarded. The above procedure was repeated five times so that a total of ten samples were collected.

The sampling site was located on a small plot of land approximately 100 feet from the base of the incinerator stack. Since the sampling site was located near a fire hydrant, it is unlikely that dumping or grading ever took place in that area. (It is possible that the rinsing of vehicles or other equipment took place in the vicinity of the fire hydrant. However, there was no evidence that this actually occurred.)

B The contents of buckets were broken apart, but were not thoroughly mixed, prior to the filling of bottles.

C Surface debris, consisting of a thin layer of dead grass, was not removed prior to insertion of the bulb planter.

D A good drawing of the sampling area was made. Distances were estimated, but were not accurately measured using a tape measure.

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(Please read Instructions on the reverse before completing)

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16. ABSTRACT

This document describes the quality assurance activities performed by Research Triangle Institute in support of Tier 4 of EPA's National Dioxin Study. Presented are the results from several technical systems audits, performance evaluation audits, and the review of relevant documentation. Conclusions are presented regarding the quality of data likely to be generated by the test program.

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